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THIS PART OF THE STUDY CENTERED UPON THE DEFINITION OF THE CRYSTALLOGRAPHY OF TWINS IN CVD DIAMOND BY MEANS OF HIGH RESOLUTION ELECTRON MICROSCOPY. FOUR BASIC KINDS OF TWINS WERE STUDIED, NAMELY: FIRST, SECOND, THIRD AND FOURTH ORDER TWINS, DESIGNATED $\Sigma=3$, $\Sigma=9$, $\Sigma=27$ AND $\Sigma=81$ RESPECTIVELY. MOST OF THE $\Sigma=3$ TWINS ARE COHERENT BUT EXAMPLE IS GIVEN OF A NON COHERENT $\Sigma=3$ ONE. THE INTERACTION BETWEEN TWINS LEAD TO THE FORMATION OF HIGHER OR LOWER ORDER ONES AND A $\Sigma=81$ TWIN HAS BEEN OBSERVED TO REDUCE THE ENERGY OF THE BOUNDARY BY TRANSFORMING TO A $\Sigma=3$ TWIN.

CONTRAST PHENOMENA RELATED TO TILTED TWINS WERE EXAMINED AND PREVIOUSLY UNSOLVED CONTRAST EXPLAINED. SEVERAL EXAMPLES OF TILTED $\Sigma=3$ AND $\Sigma=9$ TWINS ARE GIVEN. OTHER PHENOMENA RELATED TO THE CRYSTALLOGRAPHY OF TWIN QUINTUPLETS WILL BE REPORTED IN THE NEXT REPORT. IN THE FUTURE WE PLAN PLAN TO STUDY THE POSSIBILITY OF ANNEALING OUT VARIOUS TYPES OF TWIN BOUNDARIES IN DIAMOND FILMS.

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TRANSMISSION ELECTRON MICROSCOPY OF THE CVD DIAMOND FILM/SUBSTRATE INTERFACE

by

D. Shechtman

TECHNICAL REPORT TO SUBMITTED TO NIST

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SUMMARY

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INTRODUCTION

THE STRUCTURE OF GRAIN BOUNDARIES IN THE DIAMOND LATTICE, AND TWIN BOUNDARIES AS A SPECIAL CASE, RECEIVED CONSIDERABLE ATTENTION IN OVER THIRTY YEARS [1-14]. THIS IS MAINLY DUE TO THE IMPORTANCE OF DEFECTS IN SILICON AND GERMANIUM TO THEIR USEFULNESS AS EFFICIENT SEMICONDUCTORS. TWIN BOUNDARIES HAVE EQUAL IMPORTANCE TO VARIOUS PROPERTIES OF DIAMOND FILMS. ELECTRICAL AND MECHANICAL PROPERTIES AS WELL AS MIGRATION MECHANISMS ARE ALL AFFECTED BY THE TWIN BOUNDARIES AND THEIR STRUCTURE. THE EVER PRESENCE OF TWINS IN CVD DIAMOND FILMS MAKES THEIR STUDY EVEN MORE IMPORTANT COMPARED TO SEMICONDUCTORS WHICH CAN BE GROWN WITHOUT BOUNDARIES.

THE PURPOSE OF THE STUDY REPORTED HERE IS, THEREFORE, TO INVESTIGATE THE CRYSTALLOGRAPHY OF TWIN BOUNDARIES WHICH FORM IN CHEMICAL VAPOR DEPOSITED (CVD) DIAMOND FILMS, AND TO COMPARE THEM TO THE ONES WHICH WERE FOUND IN SILICON AND IN GERMANIUM. HIGH RESOLUTION ELECTRON MICROSCOPY, WHICH WAS USED IN THIS STUDY, RESOLVED THE FINE STRUCTURE OF TWIN BOUNDARIES AND THE RESULTS PRESENT A VIEW OF THE TYPE OF TWINS PRESENT IN DIAMOND THIN FILMS. OUR OBSERVATIONS INCLUDE TWIN BOUNDARIES WHICH WERE NEVER REPORTED BEFORE IN DIAMOND LATTICE MATERIALS.

TWIN BOUNDARIES IN THE DIAMOND LATTICE

TWIN BOUNDARIES CAN BE SPECIFIED BY A ROTATION AXIS, AROUND WHICH THE TWINS ARE ROTATED RELATIVE TO ONE ANOTHER, BY A ROTATION ANGLE, THE TWINING PLANE K1 AND BY THE PARAMETER Σ DEFINED AS THE RECIPROCAL OF THE DENSITY OF COINCIDENT SITES OF THE OVERLAPPING LATTICES ON BOTH SIDES OF THE BOUNDARY. THE TYPE OF BOUNDARIES REPORTED HERE INCLUDE $\Sigma=3$, $\Sigma=9$, $\Sigma=27$ AND $\Sigma=81$.

THE ROTATION AXIS

THE ROTATION AXIS OF ALL THE TWINS REPORTED HERE IS <110>. THE HIGH RESOLUTION ELECTRON MICROSCOPY MICROGRAPHS WHICH WILL BE SHOWN HAVE ALL A WELL ALIGNED <110> ZONE AXIS AND THE STUDIED TWINS ARE, THEREFORE, VIEWED EDGE-ON. THIS CONFIGURATION ALLOWS FOR A DETAILED AND RELATIVELY SIMPLE STUDY OF THE TWIN BOUNDARY'S STRUCTURAL DETAILS.

THE ROTATION ANGLE

THE ROTATION ANGLES OF DIAMOND TWINS CAN BE DETERMINED AS FOLLOWS:
FOR A $\Sigma=3$ ORIENTATION RELATIONSHIP $\alpha=2tg\sqrt{2}/2=70.52877936^\circ$
FOR A $\Sigma=9$ ORIENTATION RELATIONSHIP $\alpha=180-2[2tg\sqrt{2}/2]=38.94244126^\circ$
FOR A $\Sigma=27$ ORIENTATION RELATIONSHIP $\alpha=-180+3[2tg\sqrt{2}/2]=31.58633808^\circ$
FOR A $\Sigma=81$ ORIENTATION RELATIONSHIP $\alpha=360-4[2tg\sqrt{2}/2]=77.88488256^\circ$
THE MISMATCH ANGLE IN A TWIN QUINTUPLET $\alpha=360-5[2tg\sqrt{2}/2]=7.35610319^\circ$

DEFINITIONS:

1. **COHERENT TWIN** - CONTAINS CORRESPONDING LATTICE PLANES AND DIRECTIONS WHICH ARE CONTINUOUS ACROSS THE TWINING PLANE. MOST OF THE $\Sigma=3$ TWINS THAT WE HAVE STUDIED WERE COHERENT. THERE WERE SEVERAL EXCEPTIONS IN WHICH WE OBSERVED INCOHERENT BOUNDARIES.

2. **INCOHERENT TWIN** (ALSO CALLED LATERAL TWIN, SEMICOHERENT, NONCOHERENT OR INCOHERENT TWIN) - IS THE CASE IN WHICH THE TWIN PLANE AND THE BOUNDARY PLANE ARE NOT COINCIDENT.

3. **THE ORDER OF A TWIN BOUNDARY** - A <u>FIRST ORDER TWIN BOUNDARY</u> RESULTS FROM ONE OF THE TWINING OPERATIONS, FOR EXAMPLE: A {111} $\Sigma=3$ BOUNDARY WHICH RESULTS FROM A 70.53° ROTATION AROUND THE <110> AXIS. <u>A SECOND ORDER TWIN BOUNDARY</u> WILL FORM WHEN A TWINNING OPERATION IS PERFORMED IN A CRYSTAL DIVIDING IT, FOR EXAMPLE TO TWIN [A] AND TWIN [B]. IF TWIN [B] WILL TWIN AGAIN TO CREATE TWIN [C], AND THIS ONE WILL BE IN CONTACT WITH TWIN [A], THE BOUNDARY BETWEEN THEM IS A SECOND ORDER TWIN BOUNDARY, $\Sigma=9$. A <u>THIRD ORDER TWIN BOUNDARY</u>, $\Sigma=27$ FORMS SIMILARLY WHEN TWIN [C] TWINS AND THE RESULTANT TWIN [D] IS IN CONTACT WITH TWIN [A]. A <u>FORTH ORDER TWIN BOUNDARY</u>, FORMS AFTER FOUR STAGES OF TWINNING. KOHN [10] SUGGESTED THAT THIS TYPE OF TWIN BOUNDARY IS MUCH LIKE A HIGH ENERGY COMMON GRAIN BOUNDARY.

RESULTS

AN EXAMPLE IN WHICH FOUR KINDS OF TWINS INTERACT IS GIVEN IN FIGURE 1 [203-3]. AS IT CAN BE CLEARLY SEEN, THE CRYSTAL IS DIVIDED INTO TWO. THE UPPER PORTION HAS TWO ORIENTATIONS, DENOTED 1A AND 1B WHILE THE LOWER PORTION IS DIVIDED INTO FOUR ORIENTATIONS DENOTED 2A, 2B, 2C AND 2D. WHILE IN EACH PORTION MOST OF THE TWIN BOUNDARIES ARE OF THE Σ=3 TYPE, EXCEPT FOR THE AREA AROUND THE 2D ORIENTATION, THE BOUNDARY THAT DIVIDES THE TWO PORTIONS IS OF A HIGHER ORDER. IN ORDER TO SHOW THE SIGNIFICANT POINTS ALONG THIS BOUNDARY WE HAVE MARKED BY LETTERS THE POINT OF INTERSECTION BETWEEN IT AND THE VARIOUS Σ=3 BOUNDARIES. ALSO MARKED ARE THE {111} PLANES IN THE VARIOUS TWINS.

BETWEEN POINT A AND POINT B, A Σ=9 BOUNDARY CAN BE SEEN, EXCLUDING BRIEF ENCOUNTERS AT POINTS A' AND A" WITH Σ=3 BOUNDARIES WHICH FORM VERY NARROW TWINS. THIS BOUNDARY HAS A MISORIENTATION ANGLE OF 38.94^{o} ABOUT [110]. AT POINT A THE BOUNDARY INTERACTS WITH A Σ=3 BOUNDARY TO FORM A Σ=27 BOUNDARY WITH A MISORIENTATION ANGLE OF 31.59^{o} ABOUT [110]. ON THE LEFT HAND SIDE, AT POINT B, THE Σ=9 BOUNDARY INTERACTS WITH TWO Σ=3 BOUNDARIES TO FORM A Σ=81 BOUNDARY WITH A MISORIENTATION ANGLE OF 77.88^{o}. THIS FORTH ORDER BOUNDARY STRETCHES BETWEEN POINTS B AND C AND ONE OF ITS CHARACTERISTICS IS THE MISMATCH ANGLE OF 7.36^{o} BETWEEN THE {111} PLANES ON BOTH SIDES OF THE BOUNDARY. THIS IS CLEARLY OBSERVED ALSO BETWEEN THE INCOHERENT TWINS OF A TWIN QUINTUPLET [SEE REPORT #4, 1990]. IN THE PRESENT CASE THE MISMATCH ANGLE CLOSES AT POINT C TO FORM A LOW ENERGY Σ=3 BOUNDARY.

ANOTHER POINT THAT SHOULD BE NOTED HAS TO DO WITH THE COHERENCY OF THE Σ=3 BOUNDARIES. USUALLY THESE BOUNDARIES ARE ON THE COMMON (111) PLANE AND ARE THEREFORE COHERENT BOUNDARIES. THE Σ=3 BOUNDARIES SHOWN HERE ARE NO EXCEPTION. HOWEVER, IN ONE CASE, SEEN AT THE LOWER RIGHT PART, THE Σ=3 TWIN TILTS FROM THE COMMON (111) PLANE TO THE OTHER EDGE-ON (111) PLANE OF THE 2a ORIENTATION. THIS IS A RARE CASE, IN GENERAL, AND THE ONLY ONE IN THIS MICROGRAPH.

INCLINED TWINS

ALL THE TWIN BOUNDARIES SHOWN IN FIGURE 1 ARE ESSENTIALLY EDGE-ON, PRESENT NO CONTRAST PROBLEMS AND ENABLE AN EXAMINATION OF THE BOUNDARIES' FINE STRUCTURE. IN OTHER PARTS THE TWIN BOUNDARIES ARE FOUND TO BE IN A NON EDGE-ON POSITION WHICH RESULTS IN A CONTRAST EFFECT THAT CAN NOT BE RELATED DIRECTLY TO THE FINE STRUCTURE OF THE BOUNDARY. AN EXAMPLE OF SUCH A CASE IS GIVEN IN FIGURE 2 [202-2].

IN THIS EXAMPLE, THE UPPER PORTION OF THE MICROGRAPH HAS AN ORIENTATION DENOTED AS 1a WHILE THE LOWER PORTION IS TWINNED BETWEEN THE 2a AND 2b ORIENTATIONS AS MARKED. THE BOUNDARY THAT SEPARATES THE UPPER PORTION FROM THE LOWER ONE IS NOT EDGE-ON AND CAN BE DIVIDED INTO SECTIONS OF $\Sigma=3$ AND $\Sigma=9$ ACCORDING TO THE THE ORIENTATIONS BETWEEN 1a-2a AND 1a-2b. THE CONTRAST OBSERVED, OF THE TILTED $\Sigma=3$ AND $\Sigma=9$ IS RATHER TYPICAL, AND IT HAS BEEN OBSERVED, WITH MINOR CHANGES, AND RECORDED MANY TIMES DURING THE COURSE OF THIS STUDY. IT IS DIFFICULT TO ESTIMATE THE INCLINATION ANGLE FOR EACH BOUNDARY AT THIS POINT, BUT LATTICE SIMULATION EXPERIMENT SHOULD BE ABLE TO DEFINE IT BETTER.

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Figure 1

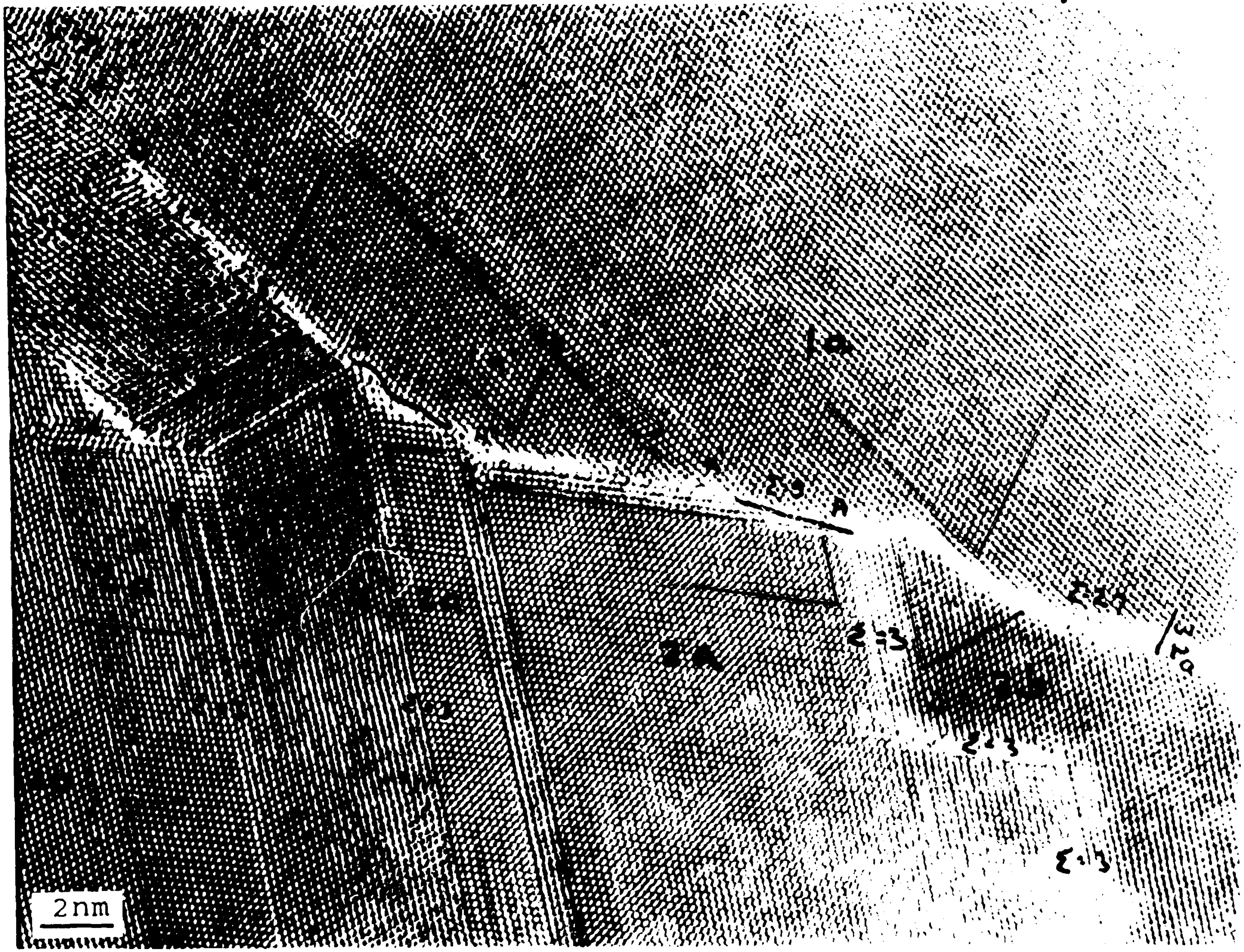
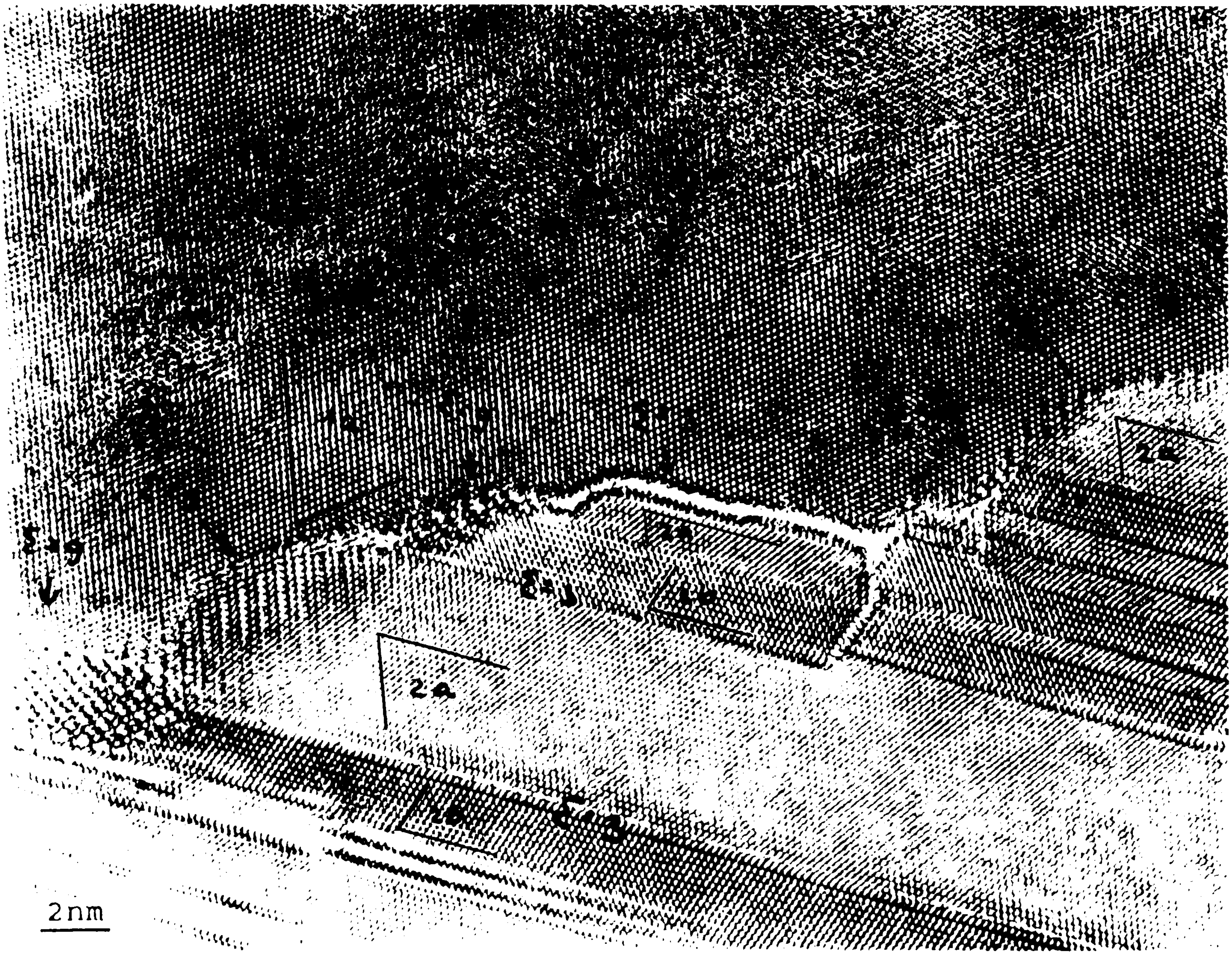


Figure 2



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